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REVISION

PROPOSED PROJECT TEST PROGRAM FOR UNIT PROCESS DESIGN CONTAMINATION TECHNICAL APPROACH

U.S.E.P.A./ERCS PROGRAM
MINDEN, WEST VIRGINIA

I. INTRODUCTION

The following test program has been developed to provide technical definition in the unit process design for the proposed soil extraction and molten salt destruction of PCB contaminated extracts at the project site in Minden, W. VA. The primary focus of the study is to enhance material handling techniques to promote continuous and consistent flows through the equipment and optimize extraction efficiency while minimizing solvent consumption. The basic unit processes are shown in figure I.

The test program has been organized into five testing categories:

1. Substrate Sizing and Clarification
2. Substrate Chemical/Physical Characterization
3. Extraction Efficiency and Contaminant Volume Reduction
4. Destruction Efficiency of the Still Bottoms
5. Environmental Impact

All testing will be done out on site using a mobile laboratory except the destruction of the still bottoms which will be done at Excell, Inc. in Newport, Ky.

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II. SAMPLE PROGRAM

The contaminated soil is presently contained in a clay-lined storage area. Ten random sample locations will be determined and soil removed from the full depth of the material. The samples will be taken using a small rubber tire backhoe and transported to a mixing area inside of the soil storage area. The individual samples will then be mixed thoroughly using the backhoe and soil removed as necessary for the test program. Prior to mixing, an aliquot will be removed from each sample and analyzed for PCB's. Samples will be analyzed as per "EPA Test Methods for Evaluating Solid Wastes," SW-846, Method 3550 (Sonication Extraction) and Method 8080 (Gas Chromatographic Method for Organochlorine Pesticides and PCB's). Two samples from the composited soil will also be removed and analyzed for PCB's.

The same sample routine will be used to collect soil from a control area at the site. One composite sample will be removed and analyzed to insure that the soil is free of PCB's. Control samples will be taken first to prevent cross-contamination.

III. SUBSTRATE SIZING AND CLASSIFICATION

The soils found at the Minden Site are typically from Regolith origin. The proposed handling process will use a series of mixing hoppers which are coupled to inclined screw auger conveyers with the extraction solvents fed counter current to the substrate flow. Since the equipment must be operated continuously to maintain the desired process schedule, the substrate particle distribution must be defined to optimize hopper and screw auger design, solvent flow rates, and solvent filtration equipment prior to recycle.

The sizing and classification tests will follow those guidelines in the ASTM method D422 (04.08), "Particle Size Analysis of Soils." Briefly, the method defines soil particle size by passing the soil through a series of particle sieves and determining the distribution based on either number of particles at each stage, and/or mass equivalent as related to the engineering needs of the project. The sieves to be used for the classification have been chosen based on visual observation of the soils and filtration of fines during solvent recycle. This is an important consideration given the density of the Freon solvent (1.565 g/cm^3). The sieve designations and sieve openings are given below:

SIEVE OR SCREEN	SIEVE OPENING, mm
2 inches	50.8
1 inch	25.4
3/4 inch	19.1
1/4 inch	6.35
No. 4	4.75
No. 10	2.00
No. 40	0.42
No. 60	0.25
No. 100	0.149

Particles in the No. 60 and No. 100 categories may be buoyant in the extraction solvent based on organic and/or water content. This buoyancy may also be transient and occur either initially due to organic or water content in discrete particles or afterwards due to agglomeration of fines by organic or water content. Therefore, the initial tests will involve immersion of the soils into the solvent prior to classification.

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TEST #1

Samples of contaminated and control soils will be obtained from the sample program (section II). Ten graduated cylinders (1000 ml) will be used for the test (5 contaminated, 5 control). 500 ml of Freon TF will be added to each cylinder. 250 grams of soil (five from each category) will then be added to each cylinder. The cylinders will be stoppered, shaken for 10 seconds, and allowed to settle. Observations will be made for each cylinder to check for settled particles, particle dispersions, buoyant particles, and emulsions. After allowing to settle for 1 minute, the solvent is slowly decanted through a tared filter paper taking care to remove only suspended and buoyant particles. The filters are allowed to air dry and reweighed to determine the gross amount of fines encountered. The soil residues left in the cylinders will be rinsed over to 1000 ml beakers, decanted, and allowed to air dry. These soils will be kept for use in the treated soil portion of sizing test.

TEST #2

Samples of the treated soils from Test #1, and additional samples of untreated soils will now be sized. The test matrices are as follows:

Freon Washed Contaminated Soils (5 Samples)	Freon Washed Control Soils (5 Samples)
Unwashed Contaminated Soils (5 Samples)	Unwashed Control Samples (5 Samples)
<u>TOTAL 20 Samples</u>	

The samples will be sized in the following order:

1. Unwashed Control Samples
2. Freon Washed Control Samples
3. Freon Washed Contaminated Samples
4. Unwashed Contaminated Samples

Samples (250g or washed residues) will be added to the sieving column and shaken until any obvious sizing has ceased. The relative amounts of each size category can be directly read off the sieving apparatus and recorded. The sieving apparatus will be disassembled and rinsed thoroughly with Freon TF between each use and allowed to dry prior to use again.

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IV. SUBSTRATE CHEMICAL/PHYSICAL CHARACTERIZATION

The chemical/physical characterization of the substrate is required to optimize solvent/substrate contact during the extraction process. The basis of this testing is focused around several perceived problems: 1) interference of the extraction solvent with excess water to form emulsions, 2) non-uniform water dispersion in the discrete soil particles to hamper penetration by the solvent and reduce extraction efficiencies, and, 3) heterogenous organic distribution in the bulk staged soils which when processed, may temporarily overload the extraction system.

The samples will be tested to determine moisture content, organic content, and inorganic content (soils). A gravimetric residue analysis will be used as outlined in "Standard Methods for the Examination of Water and Wastewater", Method 209A Total Residue Dried at 103-105°C and Method 209 E Total Volatile and Fixed Residue at 550°C. This temperature will be sufficient to volatilize the majority of organics present without promoting recombination of the PCB's into more deleterious materials.

ENERGY PROGRAM → TO SHOW IF THIS COULD HAPPEN

TEST #3

Samples for testing will be obtained from the sampling program (Section II) from the contaminated material. A 1 gram sample will be used in the test and a total of five samples will be tested. The procedure will follow those outlined in "Standard Methods" as previously cited.

Since the test soils may or may not exhibit emulsion phenomena with the extraction solvent, a series of control samples containing varied amounts of water will also be tested. This will help to define at what level the water content may begin to cause process related problems and if any drying pretreatment is needed prior to full-scale processing.

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TEST #4

Samples for testing will be obtained from the sample program (Section II) from the control material. These replicates will be run for the test. A 10 gram sample will be placed into a tared pan, heated at 550°C for 1 hour and cooled in a dessicator. The samples will be reweighed to determine the exact weight of the soil. After weighing, each group of three will be dosed with distilled water at 25%, 50%, and 100% of their weights respectively. These dosed mixtures will then be placed into 100 ml graduated cylinders containing 50 ml of Freon TF each. The cylinders will be stoppered and shaken vigorously for 10 seconds. The samples will be allowed to settle and observations made as to the formation of any emulsions or immiscible phases.

V. EXTRACTION EFFICIENCY AND CONTAMINANT VOLUME REDUCTION

This series of tests will be used primarily to define the extraction volumes and extraction durations necessary to optimize the pilot operation. Although PCB's are the compound of interest in the extraction process, preliminary testing indicates that there is a substantial amount of non-target organic material also present. Therefore the system must be designed to remove the combined organic component since the PCB's will be closely related to the distribution of the non-target organics in the soils.

The pilot-scale solvent extraction will be done as a continuous countercurrent liquid-solid extractor. The purpose for this is two-fold. First, recovery efficiency of the PCB's from the soils is contingent upon the maximization of the diffusional mass-transfer rate of the solute (PCB's) into the solvent (Freon TF). Molecular diffusivity will be optimized temporarily if the soil is repeatedly washed and drained in stages as opposed to a batch process. Secondly, continuous countercurrent liquid-solid extraction theoretically should result in a lower PCB final soil concentration than one single batch extraction due to the chemical equilibria in the process.

Due to scale requirements based on soil inventories and treatment schedules, the pilot system will be sized at 4 tons/hour. For the bench study, OHM will utilize a GREERCO continuous solid-liquid contactor which will operate at approximately 1/50 of the pilot, or 150 lbs./hr. The unit is capable of attaining solids feed rates of 0.62 - 4.9 cubic feet/hour by varying the paddle and shaft speed. As a result, retention times in the contactor can range from 2-16 minutes.

WILL GAIN ON CONTAMINANT
IN QUESTION
DOWN TIME

IS THIS AMOUNT
FOR E.P.A.
SURFACE
GOLAS

4.4
50
0.0

1 TONS
AN HOUR

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Test #5

Varying soil and fresh solvent flows through the extractor will establish PCB removal efficiencies as a function of solvent/solids ratio and retention time in the unit. For each of these runs the following samples will be withdrawn from the system and analyzed for PCB's.

Initial Soil

Soils at intermediate locations within the contactor

Soil exiting the contactor

Solvent exiting the contactor

After an optimum solvent/solids ratio and residence time has been established, the entire test procedure will be performed for three soil replicates. After processing the soil, the data will be reduced and additional testing recommended. This will include testing with 10% methanol in Freon as a second solvent to determine extraction efficiency and solvent/solids ratios.

VI. DESTRUCTION EFFICIENCY OF THE STILL BOTTOMS

The final test involves processing the still bottoms through the molten salt destruction process. Samples of the residues produced in Test #5 will be sent to the Excell, Inc. test facility in Newport, Ky. for destruction through their pilot plant. Test parameters for the destruction process will be provided by Excell.

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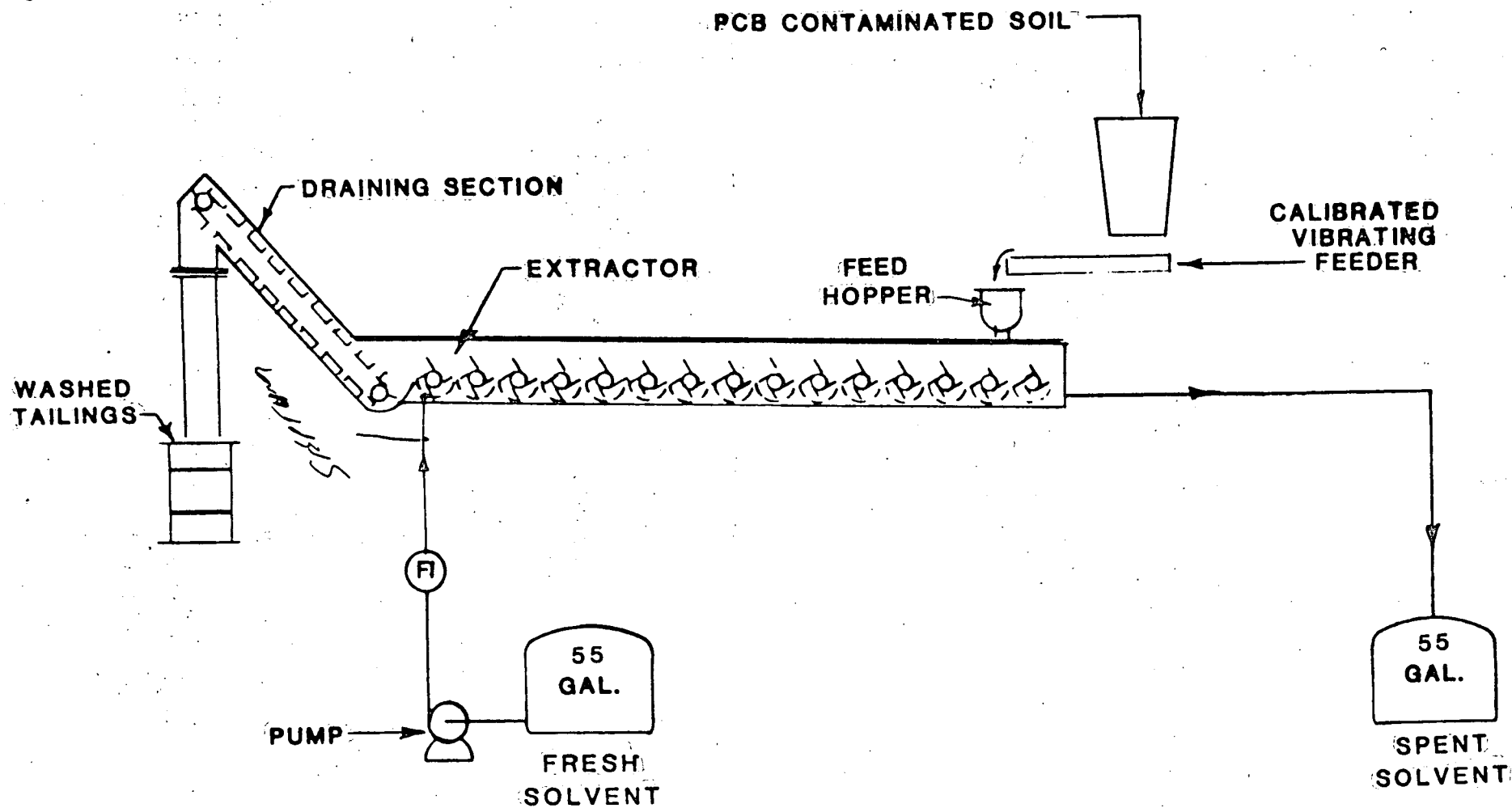
VII. ENVIRONMENTAL IMPACT

During the course of the experiment, a series of environmental analyses will be performed to monitor emissions from the ~~system~~. Continuous monitoring will be performed using an HNu ^{SYSTEM} Photoionization Detector equipped with an 11.7eV probe. This system will be operated simultaneously with the soil process equipment and all data will be recorded on stripe charts. Since the process system design is closed, it is anticipated that this data will be used primarily to assess solvent losses from the system as opposed to deleterious emissions to the environment. As a contingency measure, characterization samples will be taken for analysis by GC and/or GC/MS. The methods will include direct air collection into evacuated gas bombs and/or adsorption onto air sampling cartridges based on the concentrations determined from the real-time monitoring. One sample will be taken during each individual process test as required and all analytical methods for emission characterization will be supplied by DuPont's Freon Products Laboratory. Written documentation of these methods will be furnished at a later date.

VIII. QUALITY ASSURANCE/QUALITY CONTROL PROGRAM

All testing done during the experiment will follow the QA/QC guidelines as outlined in Section 10 of "Test Methods for Evaluating Solid Waste," USEPA, SW-846, 2nd ed., 1982 as applicable. Formal written procedures for the introduction and analysis of QA/QC samples will be furnished at the site prior to the start of the experiment.

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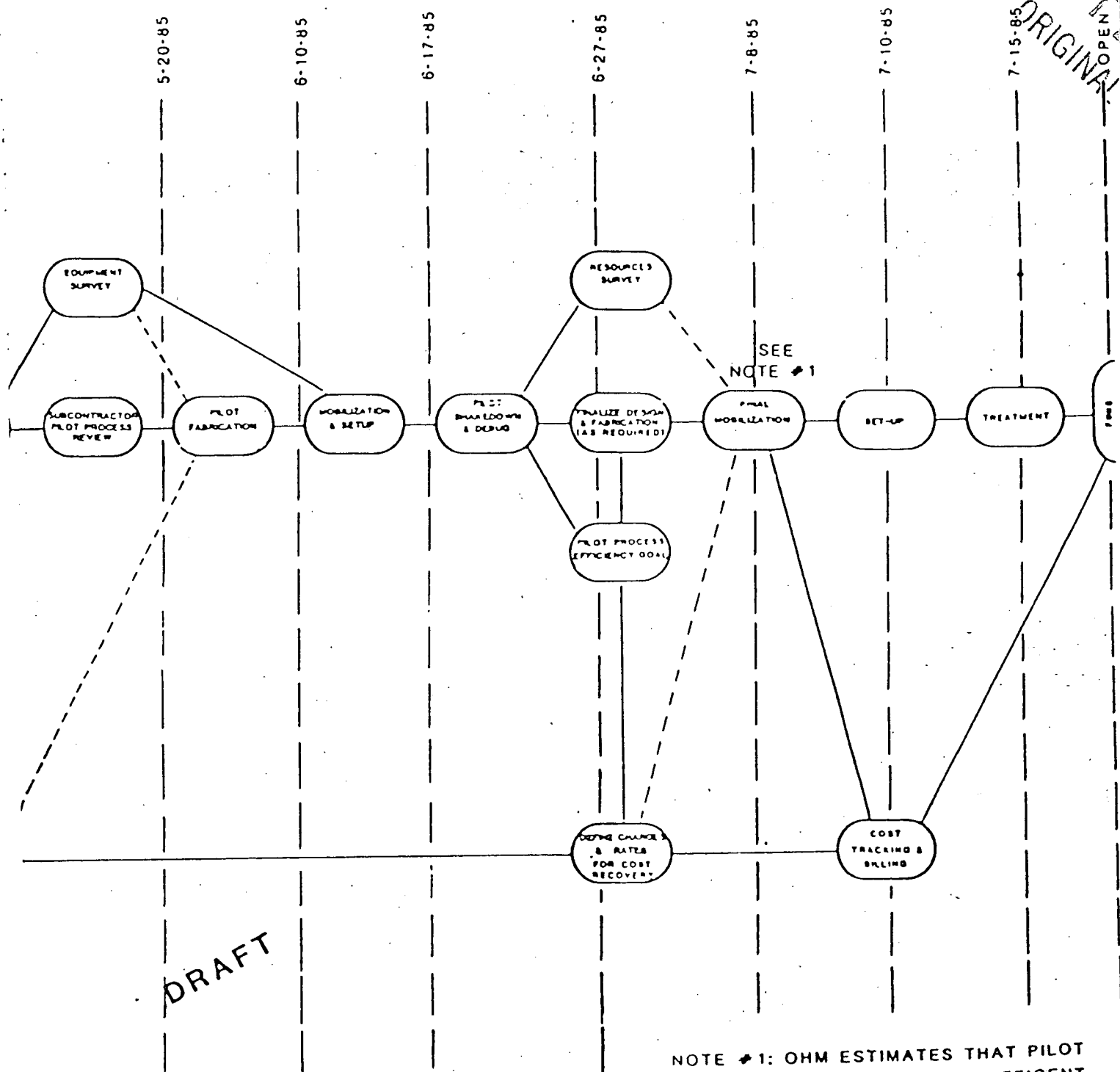
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PROPOSED SOIL EXTRACTION
FOR PCB RECOVERY
EXTRACTOR DETAIL
ERCS-US EPA REGION 3
MINDEN, WY

O. H. MATERIALS CO.
EMERGENCY RESPONSE AND
ENVIRONMENTAL RESTORATION
BOX 551 FINDLAY, OHIO
416423 3536 800 537 9540

PROJECT 2476	DRAWING A-850-2	REVISION
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NOTE #1: OHM ESTIMATES THAT PILOT SYSTEM MAY BE OF SUFFICIENT SIZE TO PERFORM THIS PROJECT.

PROJECT PLAN FOR DEVELOPMENT
& TIME SCHEDULE
PCB SOIL EXTRACTION PROCESS

O. H. MATERIALS CO.

EMERGENCY RESPONSE AND
ENVIRONMENTAL RESTORATION

BOX 551
419-423-3526

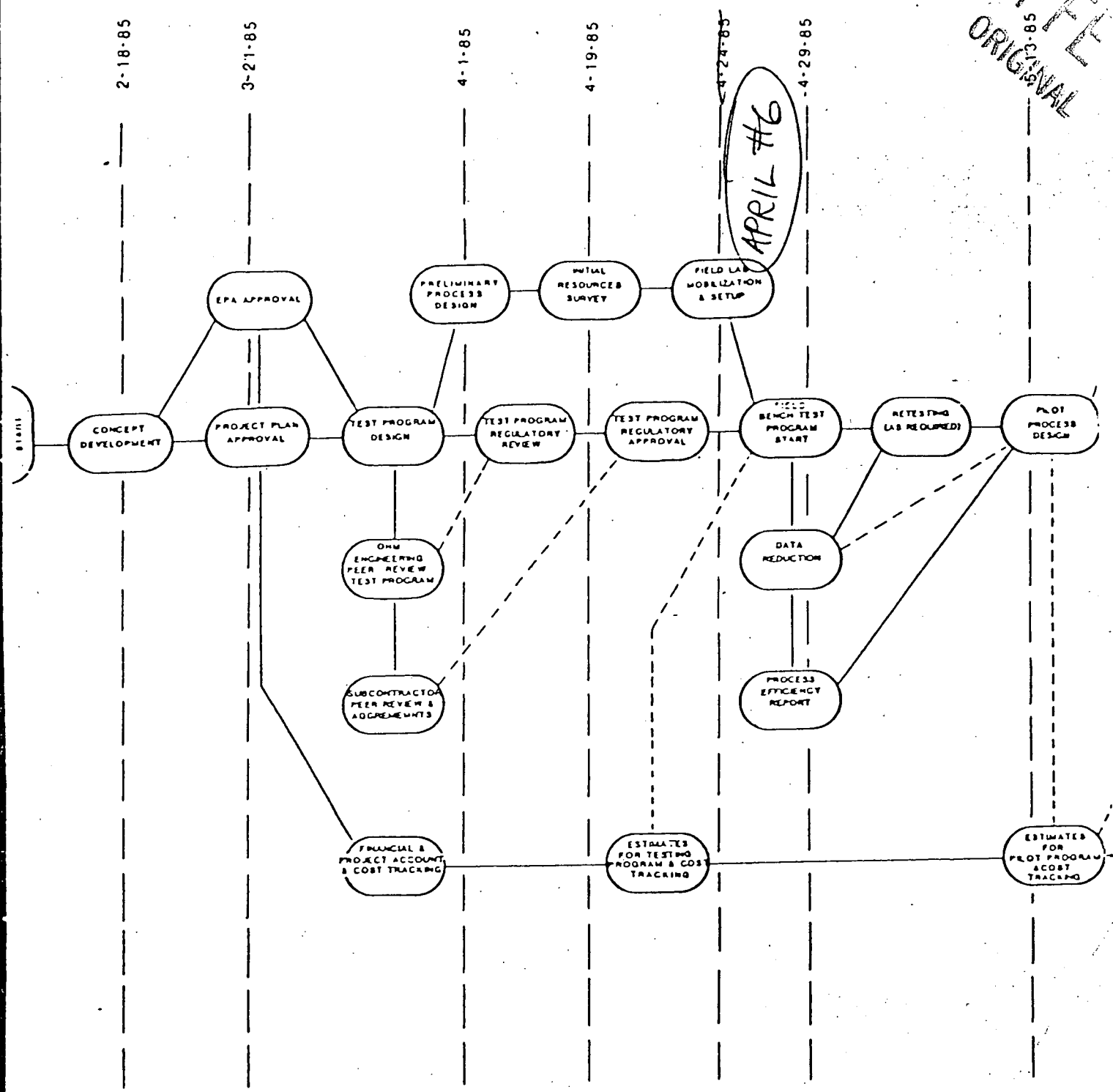
FINDLAY, OHIO
800/537-9540

MINDEN,

WEST VIRGINIA

PROJECT	DRAWING	REVISION
#2364 #2476	B-850-B	

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